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X-Ray Photoelectron Spectroscopy Study of Si-C Film Growth by  
Chemical Vapor Deposition of Ethylene on Si(100)

by

P. A. Taylor, M. Bozack, W. J. Choyke, and J. T. Yates, Jr.

Prepared for Publication in

Journal of Applied Physics

Surface Science Center  
Department of Chemistry  
University of Pittsburgh  
Pittsburgh, PA 15260

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| 1. REPORT NUMBER<br>17                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                        | 2. GOVT ACCESSION NO. | 3. RECIPIENT'S CATALOG NUMBER                                  |         |     |     |           |                 |          |     |  |
| 4. TITLE (and Subtitle)<br><br>X-Ray Photoelectron Spectroscopy Study of Si-C<br>Film Growth by Chemical Vapor Deposition of<br>Ethylene on Si(100)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |                       | 5. TYPE OF REPORT & PERIOD COVERED                             |         |     |     |           |                 |          |     |  |
| 7. AUTHOR(s)<br><br>P.A. Taylor, M. Bozack, W.J. Choyke, and<br>J. T. Yates, Jr.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              |                       | 6. PERFORMING ORG. REPORT NUMBER                               |         |     |     |           |                 |          |     |  |
| 9. PERFORMING ORGANIZATION NAME AND ADDRESS<br><br>Surface Science Center, Chem. Dept.,<br>University of Pittsburgh, Pittsburgh, PA 15260                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |                       | 8. CONTRACT OR GRANT NUMBER(s)<br><br>N00014-82-K-0280         |         |     |     |           |                 |          |     |  |
| 11. CONTROLLING OFFICE NAME AND ADDRESS                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       |                       | 10. PROGRAM ELEMENT, PROJECT, TASK<br>AREA & WORK UNIT NUMBERS |         |     |     |           |                 |          |     |  |
| 14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                       | 12. REPORT DATE<br>7/18/88                                     |         |     |     |           |                 |          |     |  |
|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |                       | 13. NUMBER OF PAGES<br>36                                      |         |     |     |           |                 |          |     |  |
|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |                       | 15. SECURITY CLASS. (of this report)<br><br>Unclassified       |         |     |     |           |                 |          |     |  |
|                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               |                       | 15a. DECLASSIFICATION/DOWNGRADING<br>SCHEDULE                  |         |     |     |           |                 |          |     |  |
| 16. DISTRIBUTION STATEMENT (of this Report)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                       |                                                                |         |     |     |           |                 |          |     |  |
| 17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |                       |                                                                |         |     |     |           |                 |          |     |  |
| 18. SUPPLEMENTARY NOTES                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       |                       |                                                                |         |     |     |           |                 |          |     |  |
| 19. KEY WORDS (Continue on reverse side if necessary and identify by block number)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                            |                       |                                                                |         |     |     |           |                 |          |     |  |
| <table> <tr> <td>Si(100)</td> <td>ISS</td> </tr> <tr> <td>CVD</td> <td>Thin film</td> </tr> <tr> <td>Silicon Carbide</td> <td>Ethylene</td> </tr> <tr> <td>XPS</td> <td></td> </tr> </table>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |                       |                                                                | Si(100) | ISS | CVD | Thin film | Silicon Carbide | Ethylene | XPS |  |
| Si(100)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       | ISS                   |                                                                |         |     |     |           |                 |          |     |  |
| CVD                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           | Thin film             |                                                                |         |     |     |           |                 |          |     |  |
| Silicon Carbide                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                               | Ethylene              |                                                                |         |     |     |           |                 |          |     |  |
| XPS                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |                       |                                                                |         |     |     |           |                 |          |     |  |
| 20. ABSTRACT (Continue on reverse side if necessary and identify by block number) The growth of a thin film of SiC grown by chemical vapor deposition (CVD) of ethylene on Si(100) at 970 K was studied by X-Ray Photoelectron Spectroscopy (XPS). The growth of the film was observed through the behavior of the Si(2p) and C(1s) core levels and their plasmon losses. A 1.2eV (towards higher binding energy) shift is observed for the Si(2p) binding energy between silicon in Si(100) and silicon in SiC. The plasmon loss energies measured as a function of film thickness below the C(1s) emission indicate that the C/Si ratio of the Si-C film throughout the CVD process is fairly constant. JTS |                       |                                                                |         |     |     |           |                 |          |     |  |

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Submitted to: J. Appl. Phys.  
Date: July 18, 1988

X-Ray Photoelectron Spectroscopy Study of Si-C Film  
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P.A. Taylor, M. Bozack<sup>†</sup>, W.J. Choyke, and J.T. Yates, Jr.

Surface Science Center,  
Department of Chemistry,  
University of Pittsburgh,  
Pittsburgh, PA 15260

<sup>†</sup>Department of Physics, Auburn University, Auburn, Alabama, 36830

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P.A. Taylor, M. Bozack<sup>+</sup>, W.J. Choyke, and J.T. Yates, Jr.

Abstract

The growth of a thin film of SiC grown by chemical vapor deposition (CVD) of ethylene on Si(100) at 970 K was studied by X-Ray Photoelectron Spectroscopy (XPS). The growth of the film was observed through the behavior of the Si(2p) and C(1s) core levels and their plasmon losses. A 1.2eV (towards higher binding energy) shift is observed for the Si(2p) binding energy between silicon in Si(100) and silicon in SiC. The plasmon loss energies measured as a function of film thickness below the C(1s) emission indicate that the C/Si ratio of the Si-C film throughout the CVD process is fairly constant.



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## 1. Introduction

Silicon carbide is a material with a wide range of uses, ranging from refractory applications, abrasive compounds, and semiconductor device material. A recent paper <sup>1</sup> describes some of the properties of silicon carbide and its potential as a semiconductor material. Growing large pure single crystals of  $\beta$ -SiC has been a difficulty impeding the use of SiC by the semiconductor industry.

A variety of growth schemes have been devised to grow thin films of SiC. Nishino and co-workers <sup>2</sup> grew up to 34  $\mu\text{m}$  thick cubic SiC single crystal films by chemical vapor deposition (CVD) on Si(100). Miyazawa <sup>3</sup> made attempts to produce 3C-SiC on Si(100) by molecular beam epitaxy, while Seaward, Barbee, and Tiller <sup>4</sup> experimented with synthesizing SiC<sub>x</sub> films with dual source sputter deposition on Si(100). Several common problems with SiC crystals grown to date involve controlling the desired polytype, the dislocation density, the impurity concentration, and the stoichiometry. The nature of the growth of Si-C films on Si(100) by CVD is the focus of this study.

Earlier work <sup>5</sup> studied the formation of SiC from an ethylene molecular beam on Si(100) at several temperatures, using XPS, AES, and ELS. This work found, by observation of the characteristic plasmon losses, that below 940 K a "Si-C alloy" was formed in which the characteristic plasmon loss at 23eV was not clearly seen. The CVD process at temperatures of 940 K or greater, or annealing the alloy, produced a Si-C film yielding the 23eV plasmon loss feature. At temperatures greater than 940 K a silicon overlayer was observed to form on top of the growing Si-C film.

The study presented here involves a careful study of the binding energy of Si(2p) and C(1s) as the Si-C layer was formed. The binding energy for silicon in the Si-C film was found to differ from that of silicon in Si(100). In addition, a study of the plasmon loss features showed that the SiC stoichiometry during the CVD process was fairly constant.

## 2. Experimental

The UHV system used in this study had a base pressure of  $1 \times 10^{-10}$  Torr; however, after repeated dosing of the crystal by  $C_2H_4$ , the pressure rose to  $6 \times 10^{-10}$  Torr and at this point the system was allowed to recover to the base pressure. The UHV system was pumped by a  $450 \text{ l s}^{-1}$  turbomolecular pump and a titanium sublimation pump (TSP) with a liquid- $N_2$  cooled jacket. The TSP jacket was kept cold throughout the CVD process.

The UHV system was equipped for XPS with Al K $\alpha$  and Mg K $\alpha$  X-ray sources, filtered by a thin aluminum foil. The Mg K $\alpha$  (1253.6eV) source was used in this study. The photoelectrons were energy analyzed with a Leybold-Heraeus EA-10 hemispherical analyzer in the  $\Delta E/E$  mode. The spectrometer resolution was calculated to be 0.9eV at 1000eV kinetic energy. The X-ray source was oriented at  $30^\circ$  to the sample plane, and the entrance optics of the energy analyzer were oriented  $90^\circ$  to the sample plane. After the CVD study was completed a silver foil (99.999%) was used to measure the spectrometer work function. The binding energy (BE) measured for the Ag(3d $_{5/2}$ ) line was 370.6eV giving a spectrometer work function of 2.7eV; (Ag(3d $_{5/2}$ ) BE = 367.9eV<sup>6</sup>). All reported energies have been corrected for the spectrometer work function.

The crystal used was a 1.5mm thick Si(100) B-doped (p-type)  $10\Omega\text{-cm}$  crystal with dimensions of  $10\times 10\times 1.5\text{mm}$ . Slots (0.4mm wide and 3mm deep) were cut into three edges of the crystal. The crystal was mounted by wedging 0.13mm thick tantalum foil (wrapped around 1.0mm diameter tungsten support wires) into two opposing slots in the crystal, as reported elsewhere <sup>5</sup>. The tungsten wires provided electrical contact for ohmic heating and thermal contact to a cooling block. The crystal was cleaned in vacuum with 3keV  $\text{Ar}^+$  ions from a differentially pumped dual plasma ion gun with a crystal current of  $2\mu\text{Acm}^{-2}$ . After sputtering the crystal was annealed to 1100 K. The sputtering and annealing cycles left traces of nickel and carbon that could not be removed from the silicon surface. The XPS intensities indicated that the surface probed had a composition of 1.8 at.% C and 0.5 at.% Ni.

During the CVD process the crystal was heated with a thyristor controlled power supply able to maintain the crystal temperature to within  $\pm 9$  K. The temperature was monitored with a 3%Re/W vs 26%Re/W thermocouple of 0.075mm diameter wedged into the third slot in the crystal. The thermocouple was calibrated once the crystal was removed from vacuum by measuring the thermocouple voltage for the phase transitions of boiling nitrogen (77 K), water-ice (273 K), boiling water (373 K), boiling glycerol (563 K), melting sodium bromide (1020 K), and melting sodium chloride (1074 K). From these calibration points the crystal temperature during the CVD process was estimated to be  $970\pm 9$  K.

The growth of the silicon carbide film was monitored by interrupting the ethylene flux at the crystal in intervals, then cooling the crystal to room temperature and digitally acquiring XPS scans. To monitor the CVD process, narrow scans of 50eV were taken for Si(2p) and

C(1s) and 20eV for Ni(2p) and O(1s). The analyzer scanned 100ms/channel for silicon and carbon and 20ms/channel for nickel and oxygen. All XPS spectra involve twenty accumulated scans, each taking 50meV steps between channels. The counts collected in a channel are reported per second, giving counts  $s^{-1}$  channel $^{-1}$ . At no time during the CVD process was oxygen detected.

The delivery of ethylene to the crystal was accomplished with a tubular doser. The tubular doser contained an internal conductance limiting orifice located between the high pressure (1-400Torr) and UHV section <sup>7</sup>. The  $C_2H_4$  flux from the end of the 1.5mm (inner) diameter tubular doser,  $f_d$  was estimated from the pressure drop in the known volume on the high pressure side of the orifice. The pressure drop was measured for three pressures in the range of 0.4-400Torr and on this basis the flux could be calculated at other  $C_2H_4$  backing pressures. The primary fluence from the doser used in the CVD process was of the order of  $f_d = 2.0 \times 10^{16}$   $C_2H_4$  molecules  $s^{-1}$  at a backing pressure of 154Torr. The  $C_2H_4$  fluence arriving at the crystal,  $f_c$ , was obtained by calculating the angular distribution of the flux from the tubular doser <sup>8</sup> and the solid angle intercepted by the crystal as described by Campbell and Valone <sup>9</sup>, accounting for the 45° tilt of the crystal on the doser axis and assuming the crystal to be circular with a 10mm diameter. These flux calculations also showed a peak  $C_2H_4$  distribution concentrated in the center of the crystal which quickly decreased toward the edges of the crystal. The fluence arriving at the crystal,  $f_c$ , was estimated to be  $f_c = (f_d)(0.7 \pm 0.1)$ . The ethylene exposure to the crystal was calculated for each  $C_2H_4$  exposure and added to the previous ethylene exposures. A Dycor M100M quadrupole mass spectrometer was used

to monitor the  $C_2H_4$  doses and the background gases. The ethylene used for the CVD process was research grade gas (99.95%) from Airco, further purified by a series of freeze-pump-thaw cycles.

The differentially pumped plasma ion gun had the capability to deliver a focused beam, diameter = 1mm, of  $He^+$  for ion scattering spectroscopy (ISS). The hemispherical analyzer, in the  $\Delta E$  mode, was used to energy analyze the scattered  $He^+$  ions. ISS was performed with 1000eV  $^3He^+$  ions with an angle of  $123^\circ$  between the ion gun and the scattered ions collected by the energy analyzer.

### 3. Results

The scans of the Si(2p) region and the C(1s) region from the sputtered and annealed silicon surface are shown in Fig. 1a and 1b respectively. The  $^3He^+$  ISS from the same clean surface is shown in Fig. 1c. The ISS shows a Si peak and a low energy loss peak due to multiple energy losses of  $^3He^+$ . The limit of detection of surface Ni for  $^3He^+$  estimated from the noise level at  $E/E_0 = 0.854$  and sensitivity factors<sup>10</sup> was calculated to be approximately 0.4 at.% Ni. XPS measurement of the Ni(2p) feature at a binding energy of 855.6eV indicated a concentration of  $\sim 0.5$  at.% Ni in the volume of Si probed. If the Ni was concentrated at the surface and not evenly distributed in the volume probed by XPS, the expected Ni ISS signal would be approximately one third of the Si signal. Since no Ni was observed, these calculations suggest that the surface concentration of Ni was negligible and that Ni played no role in the CVD process.

The elemental sensitivity factor (ESF) method<sup>11</sup> was used to obtain a quantitative measure of the surface impurities and the carbon

uptake during the CVD process. To obtain the ratio of carbon (or nickel) to silicon the ESF method states that

$$\frac{N_a}{N_b} = \frac{I_a \sigma_b \lambda_b(KE) T_b(KE)}{I_b \sigma_a \lambda_a(KE) T_a(KE)} \quad (1)$$

where  $N_a$  corresponds to the number of atoms of type 'a', and  $I_a$  is the area under the XPS line with a linear background subtracted. The photoionization cross-sections,  $\sigma$ , were taken from Scofield<sup>12</sup> and corrected for asymmetry of photoemission from different orbitals<sup>13</sup> and elastic scattering of the photoelectron traveling through matter<sup>14</sup>. The inelastic mean free path of the photoelectron,  $\lambda(KE)$ , was calculated according to the work of Penn [15]. The transmission efficiency of the energy analyzer,  $T(KE)$ , was taken to be  $(KE)^{-1}$  for the energy analyzer in the  $\Delta E/E$  mode. The values used for  $\sigma$ ,  $\lambda(KE)$ , and  $T(KE)$  are given in Table I.

In Fig. 2 the uptake of carbon by the Si(100) crystal at 970 K is shown as a function of the number of  $C_2H_4$  molecules arriving at the crystal. The general shape of the carbon uptake curve is similar to that previously reported for the CVD process using  $C_2H_4$  on Si(100) in this temperature range<sup>5</sup>, although a factor of approximately 10 difference in exposure exists for the break point in this curve ( $\sim 2.5 \times 10^{20} C_2H_4$ ) and in our previous studies in a different apparatus ( $\sim 2.5 \times 10^{19} C_2H_4$ ).

The carbon to silicon ratio maxima obtained from Fig. 2 indicates that the silicon carbide formed by CVD at 970 K has a stoichiometry of  $SiC_{0.6}$ . However, the behavior of the plasmon loss feature, discussed below, indicates a stoichiometry closer to SiC. The discrepancy in

stoichiometry may be due to assumptions in the ESF quantitative analysis or due to the presence of a silicon overlayer <sup>5</sup>. On the basis of the plasmon loss features, the silicon carbide film grown here is taken to be Si-C (1:1).

As the Si-C layer forms, a new Si(2p) photoelectron feature is observed  $1.2 \pm 0.2$  eV higher in binding energy than the overlapping Si(2p) features from clean Si(100), as shown in Fig. 3a. This new feature is assigned to the photoelectron from the Si(2p) level in Si-C. Only one C(1s) photoelectron feature is observed during the CVD process (Fig. 3b), and this feature grows in intensity with increasing exposure of C<sub>2</sub>H<sub>4</sub> to the Si(100) crystal. The binding energy of the C(1s) peak decreases by 0.3 eV throughout the CVD process and has an average value of  $285.6 \pm 0.2$  eV over the range of observation. Both the Si(2p) and the C(1s) binding energies are within the range of previously reported binding energies (see Table II), with our values for C(1s) and Si(2p) being on the high side of the mean of all the values reported.

The formation of the Si-C film is also observed by the behavior of the plasmon loss features, as shown in Fig. 4. In figure 4a, the curve 'a' shows the silicon plasmon loss feature at 17.2 eV and the double loss at 34.5 eV observed for Si(100). As the Si-C film is grown, a new plasmon loss feature is observed at 22.7 eV (Fig. 4a curves e-h). The plasmon loss at 22.7 eV is attributed to the energy loss experienced by the Si(2p) photoelectron as it excites a plasmon in SiC <sup>5,16</sup>. A plasmon loss feature at 22.7 eV is also observed, associated with the C(1s) photoelectron feature (Fig. 4b).

<sup>3</sup>He<sup>+</sup> ISS performed throughout the CVD process, after cooling to 298 K, showed only a peak for Si. The Si ISS peak decreased in inten-

sity as the SiC film grew, as shown in Fig. 5 and 6. In Fig. 6, it may be seen that the decrease in the ISS signal exhibits a breakpoint at  $\sim 2 \times 10^{20}$  C<sub>2</sub>H<sub>4</sub> molecules incident; this is a very similar behavior to that observed by XPS, with a breakpoint at  $\sim 2.5 \times 10^{20}$  C<sub>2</sub>H<sub>4</sub> incident.

The only other loss feature was the peak due to multiple He<sup>+</sup> collisions and/or secondaries. At no time during the CVD process was carbon, nickel, or oxygen observed by <sup>3</sup>He<sup>+</sup> ISS. The lack of a carbon loss feature is not surprising considering the low sensitivity to carbon ( $s_C = 0.29$ ,  $s_{Si} = 2.1$ ,  $s_{Ni} = 12.1$  as reported for 2keV <sup>3</sup>He<sup>+</sup>)<sup>10</sup>; in addition there exists the possibility of shadowing of the carbon by the larger silicon atoms <sup>17</sup> or the presence of an overlayer of silicon on the SiC film <sup>5</sup>.

#### 4. Discussion

##### 4.1 Nature of the Si-C Film

The binding energies of C(1s) and Si(2p) from SiC and their respective plasmon loss features show little or no shift ( $\Delta E_B < 0.5\text{eV}$ ) in peak maxima during the CVD process (see Fig. 7 and 4). This lack of shift differs from the monotonic shift observed for the Si(2p) peak and the plasmon loss features in Si<sub>1-x</sub>C<sub>x</sub>:H amorphous films grown as a function of varying 'x' <sup>26</sup>. The amorphous Si<sub>1-x</sub>C<sub>x</sub> films were grown on silicon by reactive sputtering of polycrystalline silicon and graphite targets with an Ar-H<sub>2</sub> gas mixture. Si<sub>1-x</sub>C<sub>x</sub>:H films were made with varying values of 'x' and studied by XPS <sup>26</sup>. A monotonic increase in the binding energy of the Si(2p) and C(1s) core electrons was observed for increasing values of 'x' with an range of 2.0eV for Si(2p) and a 1.3eV

for C(1s) <sup>19</sup>. The plasmon loss features exhibited a monotonic 5.5eV shift between values of  $x = 0$  ( $\hbar\omega_p = 17.2\text{eV}$ ) and  $x = 0.55$  ( $\hbar\omega_p = 22.7\text{eV}$ ) <sup>26</sup>.

The small monotonic shifts of  $\sim 0.5\text{eV}$  for C(1s)<sub>SiC</sub> (Fig.7) and  $\sim 0.3\text{eV}$  for Si(2p)<sub>SiC</sub> binding energy (Fig. 7) in the CVD process studied here, suggest that the silicon and the carbon stoichiometry in the SiC film does not vary greatly as the film grows thicker. The plasmons, however, offer a better measure of change in bulk stoichiometry since the plasmon energy,  $\hbar\omega_p$ , depends for semiconductors on the density the valence electrons <sup>27</sup>. Therefore, the small monotonic shift of  $\sim 1.9\text{eV}$  observed associated the plasmon loss feature for the C(1s) peak (see Fig.4b) indicates that only a small change in stoichiometry within the thin film takes place in CVD-grown Si-C film.

#### 4.2 Efficiency of SiC Production by CVD at 970 K

The efficiency of SiC production by ethylene CVD on Si(100) can be estimated by using a layer model in which SiC layers grow on the Si(100) <sup>5</sup>. To calculate the efficiency of SiC production, the ratio of carbon and silicon XPS intensities,  $I[\text{C}(1s)]/I[\text{Si}(2p)]$ , are calculated for an increasing number of SiC layers. The carbon-to-silicon XPS intensity ratio is given by

$$\frac{I[\text{C}(1s)]}{I[\text{Si}(2p)]} = \frac{I_{\text{c}}^{\text{SiC}}}{[I_{\text{Si}}^{\text{SiC}} + I_{\text{Si}}^{\text{Si}}]} \quad (2)$$

where the carbon signal originates from the SiC and the silicon signal from both the SiC layers and the underlying Si substrate. The indivi-

dual terms in above expression are given by

$$I_C^{SiC} = \sigma_C N_C^{SiC} \sum_{i=0}^{n-1} \exp[-(2i) d_{SiC} / \lambda_C^{SiC}] \quad (3)$$

$$I_{Si}^{SiC} = \sigma_{Si} N_{Si}^{SiC} \sum_{j=0}^{n-1} \exp[-(2j+1) d_{SiC} / \lambda_{Si}^{SiC}] \quad (4)$$

$$I_{Si}^{Si} = \sigma_{Si} N_{Si}^{Si} \sum_k^{\infty} \exp[-k d_{Si} / \lambda_{Si}^{Si}] \exp[-2n d_{SiC} / \lambda_{Si}^{SiC}] \quad (5)$$

where  $\lambda_{Si}$  and  $\sigma$  ( $\sigma = \sigma_{relative}$ ) are given in Table II. The electron mean free path of the Si(2p) electron in silicon, excited by the Mg Ka source is  $\lambda_{Si} = 26.8\text{\AA}$ , and  $N_{SiC} = 1.05 \times 10^{15}$  C or Si atoms in a  $1\text{-cm}^2$  layer of cubic SiC assumed to form on Si(100) <sup>2</sup> of thickness  $d_{SiC}$ . Here the thickness of SiC and Si layers were taken to be  $d_{SiC} = 1.089\text{\AA}$  and  $d_{Si} = 1.357\text{\AA}$  respectively, calculated from their lattice constants (Si<sup>37</sup>, SiC<sup>38</sup>).

From the above model and assuming several efficiencies of SiC production,  $I[C(1s)]/I[Si(2p)]$  intensity ratios were calculated and compared with experimental intensity ratios as shown in Fig. 8. From Fig. 8 an efficiency of  $\eta = 2 \times 10^{-5}$  SiC units is obtained for every  $C_2H_4$  collision with the Si(100) surface at 970 K. A sharp deviation from model is seen in the experimental data at  $\sim 3 \times 10^{20}$  incident  $C_2H_4$  molecules on the crystal. This deviation must be due to some mechanism limiting the SiC growth after a certain SiC thickness is achieved. The

limiting mechanism may be the diffusion of Si to the surface which would react with the gas phase  $C_2H_4$ .

This efficiency may be compared to that estimated on the basis of the attenuation of the  $Si(2p)_{Si}$  XPS intensity. Assuming the attenuation of the  $Si(2p)_{Si}$  XPS intensity is solely due to SiC layers on the Si substrate the final-to-initial silicon intensity,  $I^f[Si(2p)_{Si}]/I^o[Si(2p)_{Si}]$ , is given by

$$I^f[Si(2p)_{Si}]/I^o[Si(2p)_{Si}] = \exp(-X_{SiC}/\lambda_{Si}^{Si}) \quad (6)$$

where  $X_{SiC}$  is the thickness of the SiC film and  $\lambda_{Si}^{Si} = 22.2\text{\AA}$ . Using the experimental integrated areas, the intensity ratio is  $I^f[Si(2p)_{Si}]/I^o[Si(2p)_{Si}] = 0.259$  where the final intensity of  $Si(2p)_{Si}$  was taken at  $2.2 \times 10^{20}$   $C_2H_4$  incident molecules (the breakpoint of initial constant uptake of carbon). An estimated 14 layers of SiC were produced with the crystal exposed to  $2.2 \times 10^{20}$   $C_2H_4$  molecules. Accounting for two SiC units/ $C_2H_4$ , an efficiency of  $\eta = 3.3 \times 10^{-5}$  SiC units are produced per  $C_2H_4$  collision with the Si(100) surface, at 970 K, using this second method to estimate the SiC layer thickness. These two methods for calculating the efficiency of SiC formation agree well within this experiment, but differ by a factor of  $\sim 10^{-2}$  from previously calculated efficiencies for SiC production from ethylene CVD growth on Si(100) at 940 K <sup>5</sup>.

## 5. Conclusions

These results indicate that the growth of Si-C films by chemical vapor deposition of ethylene on Si(100) at 970 K occurs in such a

fashion that the stoichiometry of the Si-C film is fairly constant throughout the CVD process. The C/Si ratio in the Si-C film was observed through both the behavior of the Si(2p) and C(1s) core electron binding energies and the plasmon loss features. The binding energy of the Si(2p) electrons differ by 1.2eV between silicon in Si(100) and Si-C, where the silicon in the Si-C film has a higher binding energy. The calculated initial efficiency of SiC production from ethylene CVD at temperatures between 940 - 970 K is of the order of  $10^{-4}$  -  $10^{-5}$  SiC units produced for every C<sub>2</sub>H<sub>4</sub> collision with the Si(100) surface at 970 K. The film growth efficiency is greatly reduced once a film thickness of 30Å is achieved. At this thickness some mechanism, such as diffusion, limits further Si-C film growth.

## 6. Acknowledgement

The authors acknowledge with thanks the support of this work by the Office of Naval Research (ONR) under contract No. N00014-82-K-0280.

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Cross-sections are given relative to C(1s) with a cross-section of 22,200 barns at 1254eV.

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### Figure Captions

Figure 1. XPS-ISS spectra of clean Si(100). A,B) XPS of clean Si(100); C) ISS of clean Si(100).

Figure 2. XPS determination of Carbon uptake by Si(100) during C<sub>2</sub>H<sub>4</sub> CVD film growth.  $N_C/N_{Si}$  determined by elemental sensitivity factor (ESF) analysis of XPS data.

Figure 3. Behavior of Si(2p) and C(1s) XPS features during Si-C film growth at 970 K. A) Si(2p)<sub>Si</sub> intensity decreases in intensity as the SiC film grows and Si(2p)<sub>Si</sub> intensity increases. B) C(1s)<sub>SiC</sub> feature. Curves correspond to 0.0, 0.73, 1.27, 1.67, 2.20, 3.00, and  $7.4 \times 10^{20}$  C<sub>2</sub>H<sub>4</sub> molecules incident on the crystal.

Figure 4. Development of plasmon loss features observed by XPS during Si-C film growth on Si(100) at 970 K. A) plasmon losses from Si(2p) XPS line, where  $\hbar\omega_p(Si) = 17.2\text{eV}$  and  $\hbar\omega_p(SiC) = 22.7\text{eV}$ . B) Plasmon losses from C(1s) XPS line. The SiC plasmon loss energy,  $\hbar\omega_p(SiC)$ , shifts during film grows over a  $\sim 1.9\text{eV}$  range. Curves b-h correspond to 0.73, 1.27, 1.67, 2.20, 3.00, 3.80, and  $7.4 \times 10^{20}$  C<sub>2</sub>H<sub>4</sub> molecules incident on the crystal.

Figure 5. ISS behavior during Si-C film growth on Si(100) at 970 K. Throughout the CVD process only the Si loss feature is observed. Curves correspond to a:0.0, c:1.27, e:2.20, and h: $7.4 \times 10^{20}$  C<sub>2</sub>H<sub>4</sub>.

Figure 6. Decay of the Si ISS signal during  $C_2H_4$  CVD Si-C film growth on Si(100) at 970 K.

Figure 7. Shift in the  $Si(2p)_{SiC}$  and  $C(1s)_{SiC}$  binding energies during  $C_2H_4$  CVD Si-C film growth on Si(100) at 970 K.

Figure 8. Efficiency of carbon uptake as measured by the  $C(1s)/Si(2p)$  XPS intensity ratios during  $C_2H_4$  CVD Si-C film growth on Si(100) at 970 K. Open symbols correspond to model layer calculations and filled symbols to experimental data. An initial efficiency of  $\eta \approx 2 \times 10^{-5}$   $C_2H_4$  molecules react to form SiC per  $C_2H_4$  collision. The experimental data deviates from the model at  $\sim 3 \times 10^{20}$   $C_2H_4$  molecules incident on the crystal.

Table I. Parameters for Elemental Sensitivity Factor Analysis

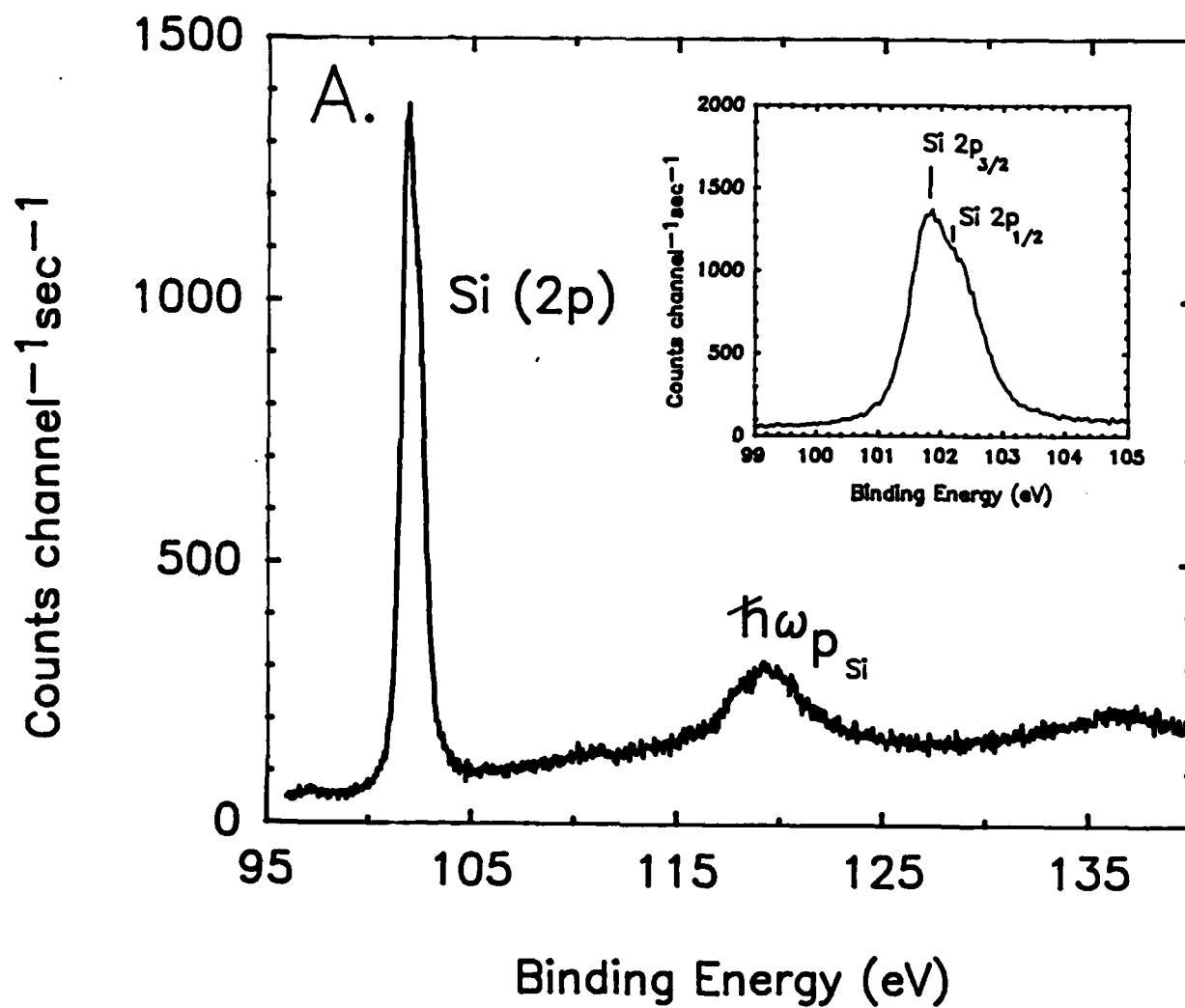
| Parameter                                                                      | Si(2p)               | C(1s)                | Ni(2p)               | Reference |
|--------------------------------------------------------------------------------|----------------------|----------------------|----------------------|-----------|
| Scofield cross-section<br>$\sigma_{\text{relative}}$                           | 0.865                | 1.00                 | 21.11                | 12        |
| Cross-section corrected<br>for asymmetry and<br>elastic scattering, $\sigma_a$ | 0.816                | 0.982                | 19.80                | 13, 14    |
| Inelastic mean free<br>path, $\lambda(\text{KE})$                              | 22.2 Å               | 19.2 Å               | 9.7 Å                | 15        |
| Transmission<br>efficiency, T(KE)                                              | $8.7 \times 10^{-4}$ | $1.0 \times 10^{-3}$ | $2.5 \times 10^{-3}$ |           |

Table II. Reported Binding Energies for SiC

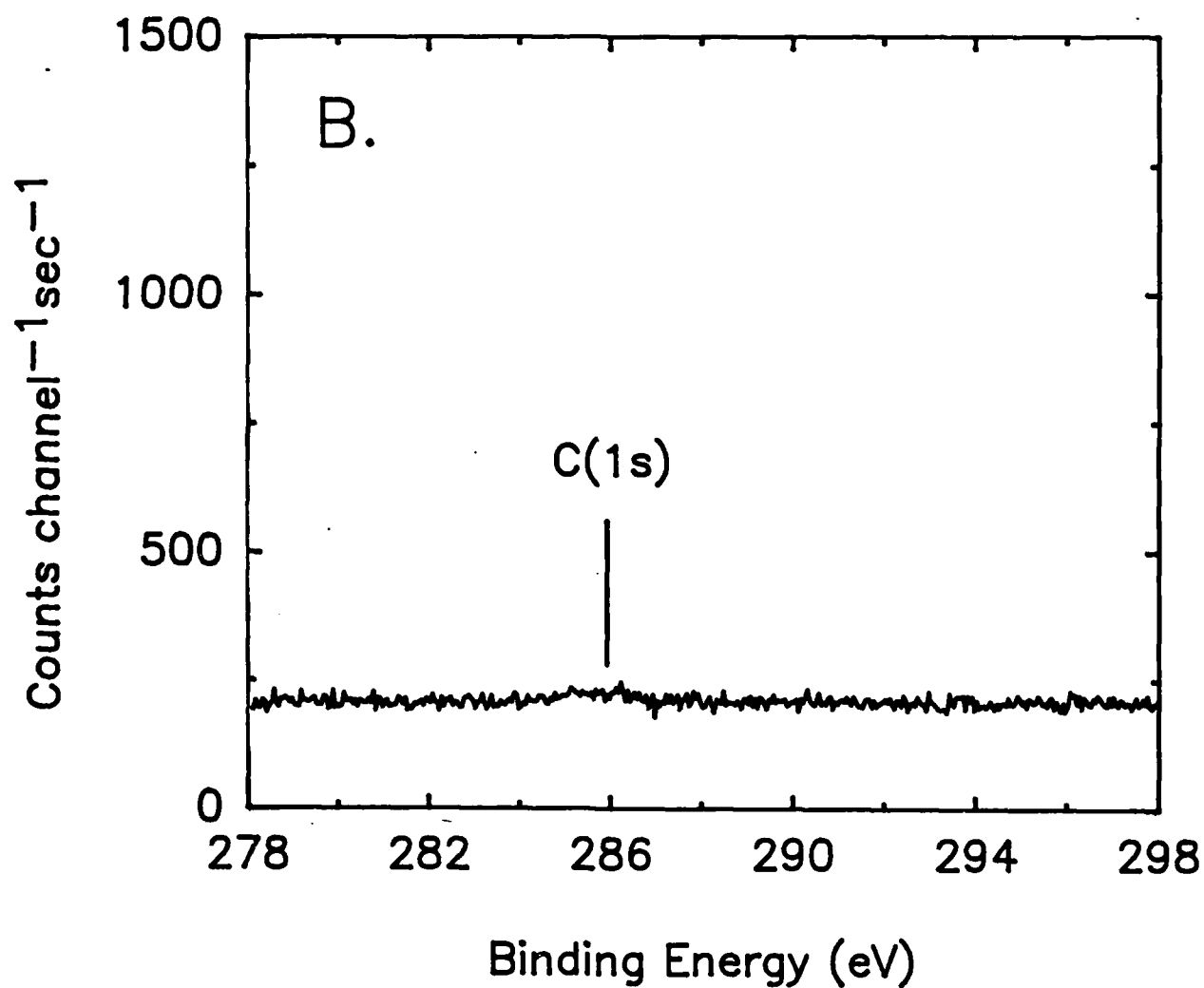
| Nature of SiC        | Si(2p), eV | C(1s), eV | Standard               | Reference |
|----------------------|------------|-----------|------------------------|-----------|
| CVD Grown film       | --         | 280.7     | None given             | 5         |
| single crystal       | 103.6      | 286.1     | None given             | 16        |
| powder               | 102.2      | --        | hydrocarb. contam.     | 28        |
| React. Sputter SiC:H | 98.8       | --        | Ag(4f <sub>7/2</sub> ) | 19        |
| Glow Discharge SiC:H | 99.6       | --        | Ar(1s)                 | 29        |
| single crystal       | 100.5      | 282.9     | Fermi Level            | 30        |
| CVD Grown film       | 100.5 (a)  | 283.4     | Au(4f <sub>7/2</sub> ) | 31        |
| single crystal       | 100.5      | 282.7     | Au(4f <sub>7/2</sub> ) | 32        |
| single crystal       | 98.97      | 282.26    | None given             | 33        |
| single crystal       | 100.4      | 283.6     | Au(4f <sub>7/2</sub> ) | 34        |
| CVD Grown film       | 103.3      | 285.6     | Ag(3d <sub>5/2</sub> ) | this work |
| polycrystal          | 100.2      | 283.2     | Au(4f <sub>7/2</sub> ) | 35        |
| not stated           | 103.9      | --        | Fermi Level            | 36        |

(a) Si(2p) calculated from Si(2s) line reported.

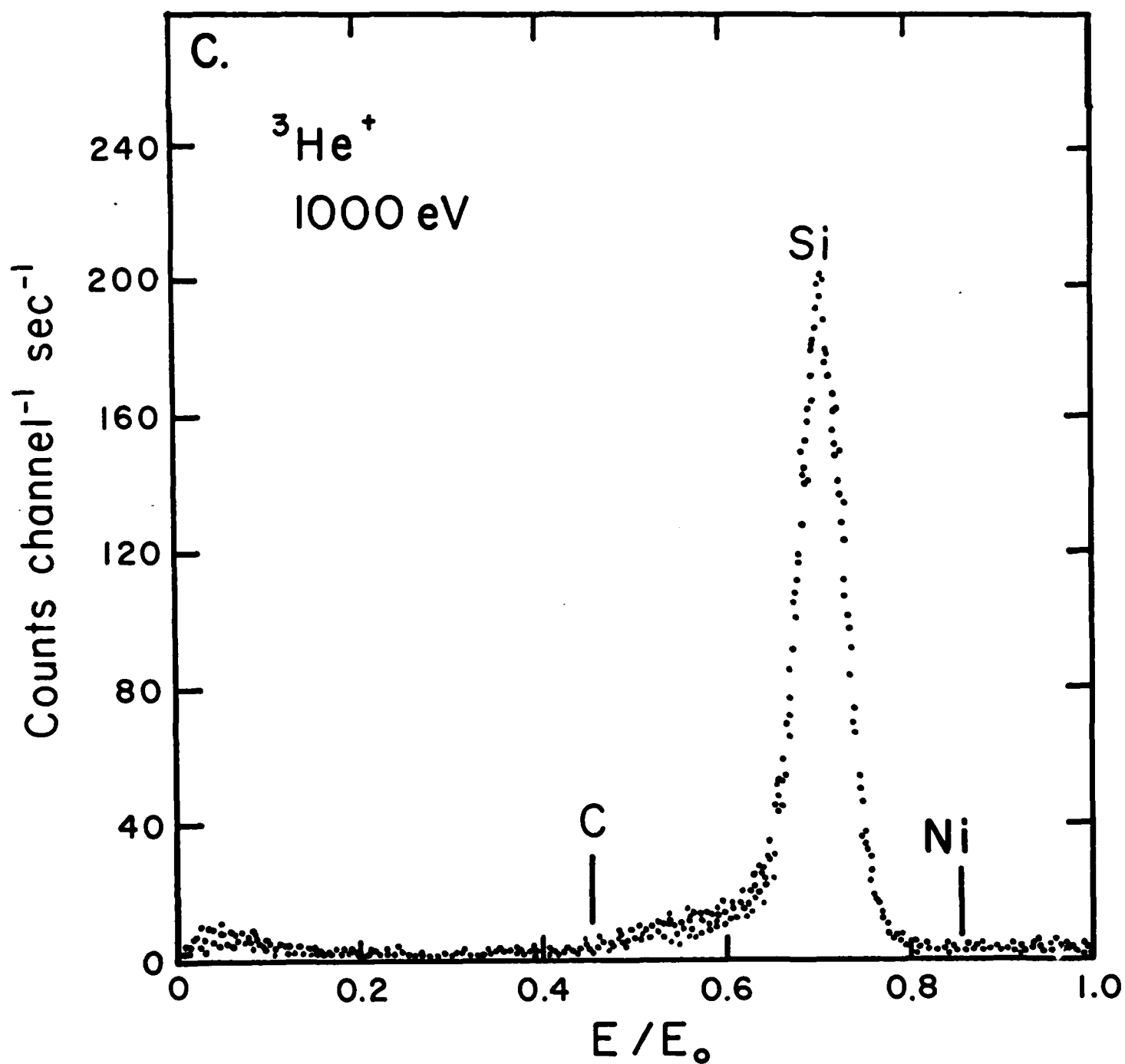
## XPS of Clean Si(100) Surface



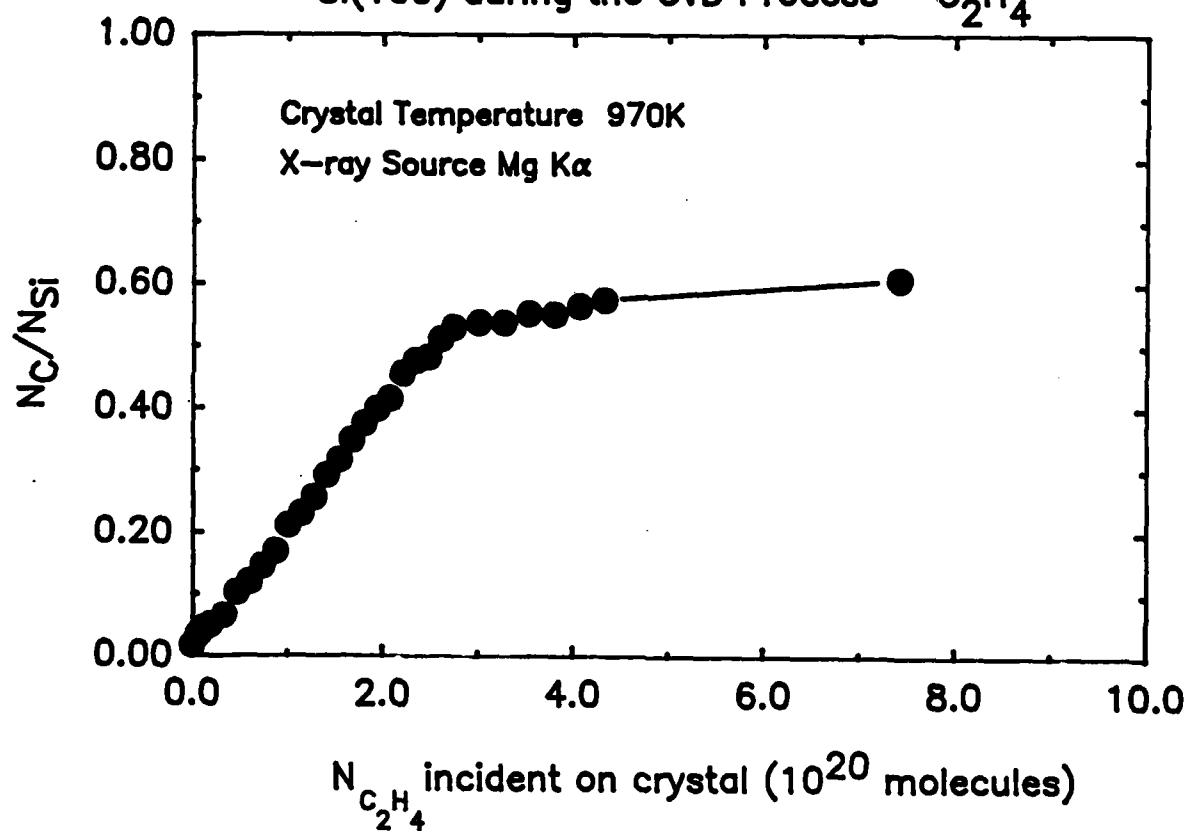
## XPS of Clean Si(100) Surface



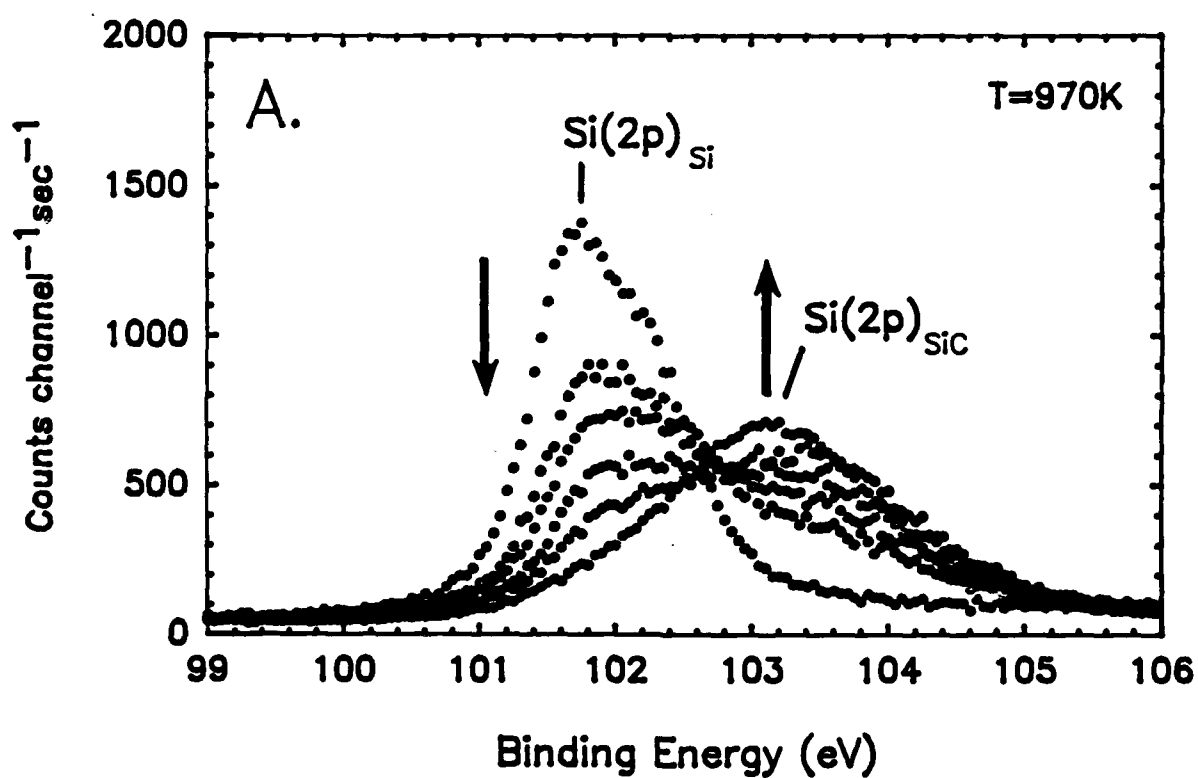
# $^3\text{He}^+$ ISS of Clean Si(100)

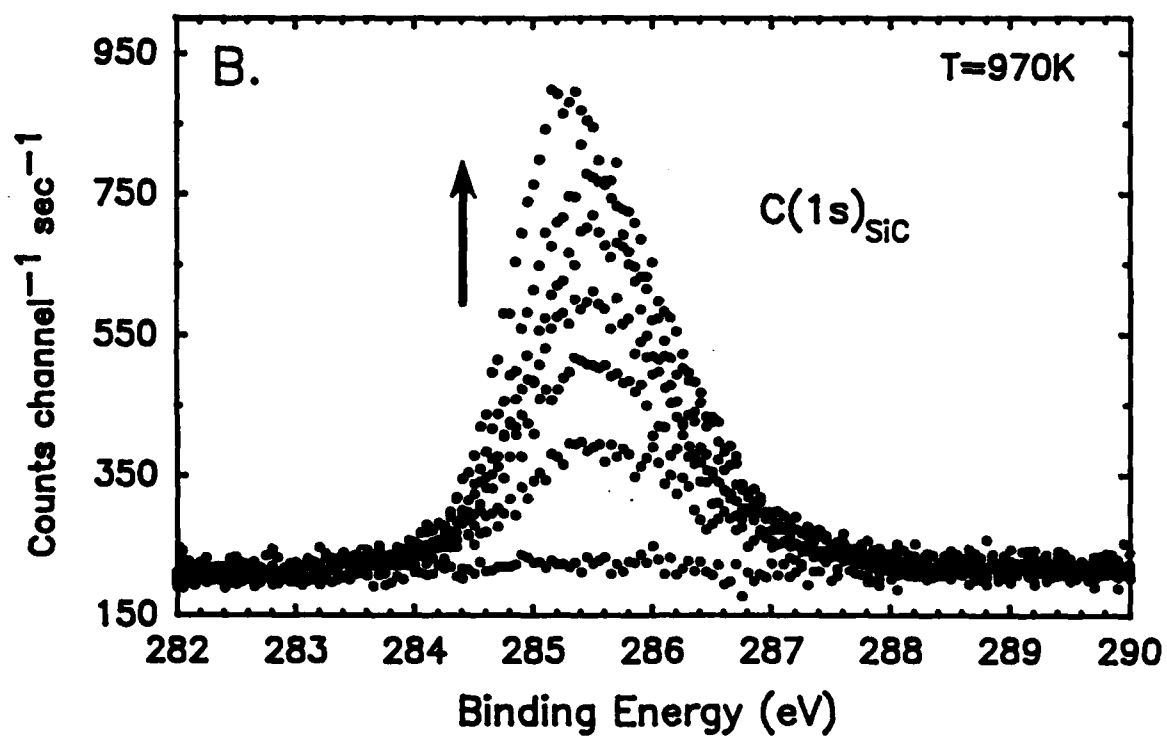


XPS Determination of the Carbon Uptake by  
Si(100) during the CVD Process -  $C_2H_4$

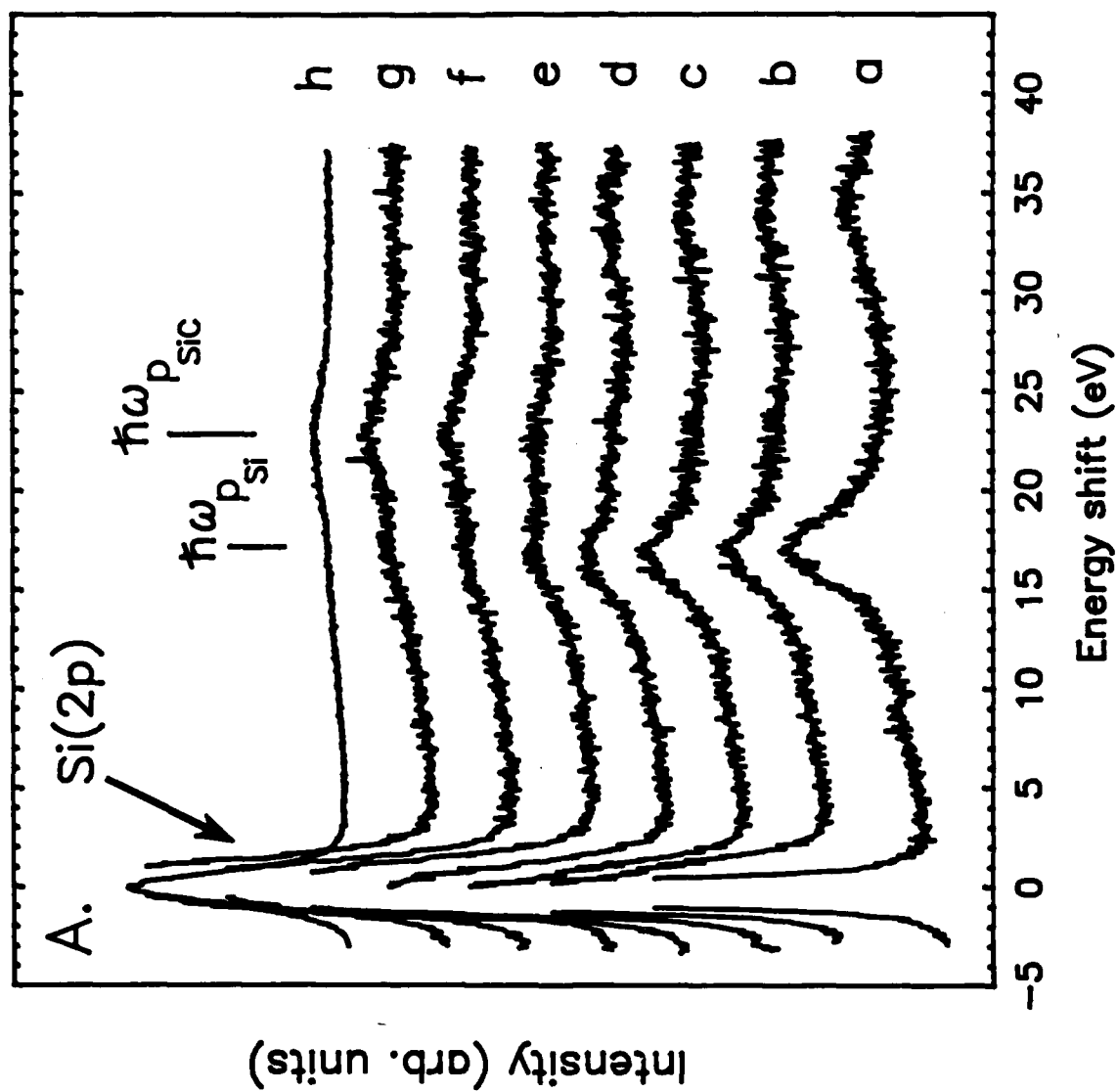


# XPS Behavior During Film Growth on Si(100)

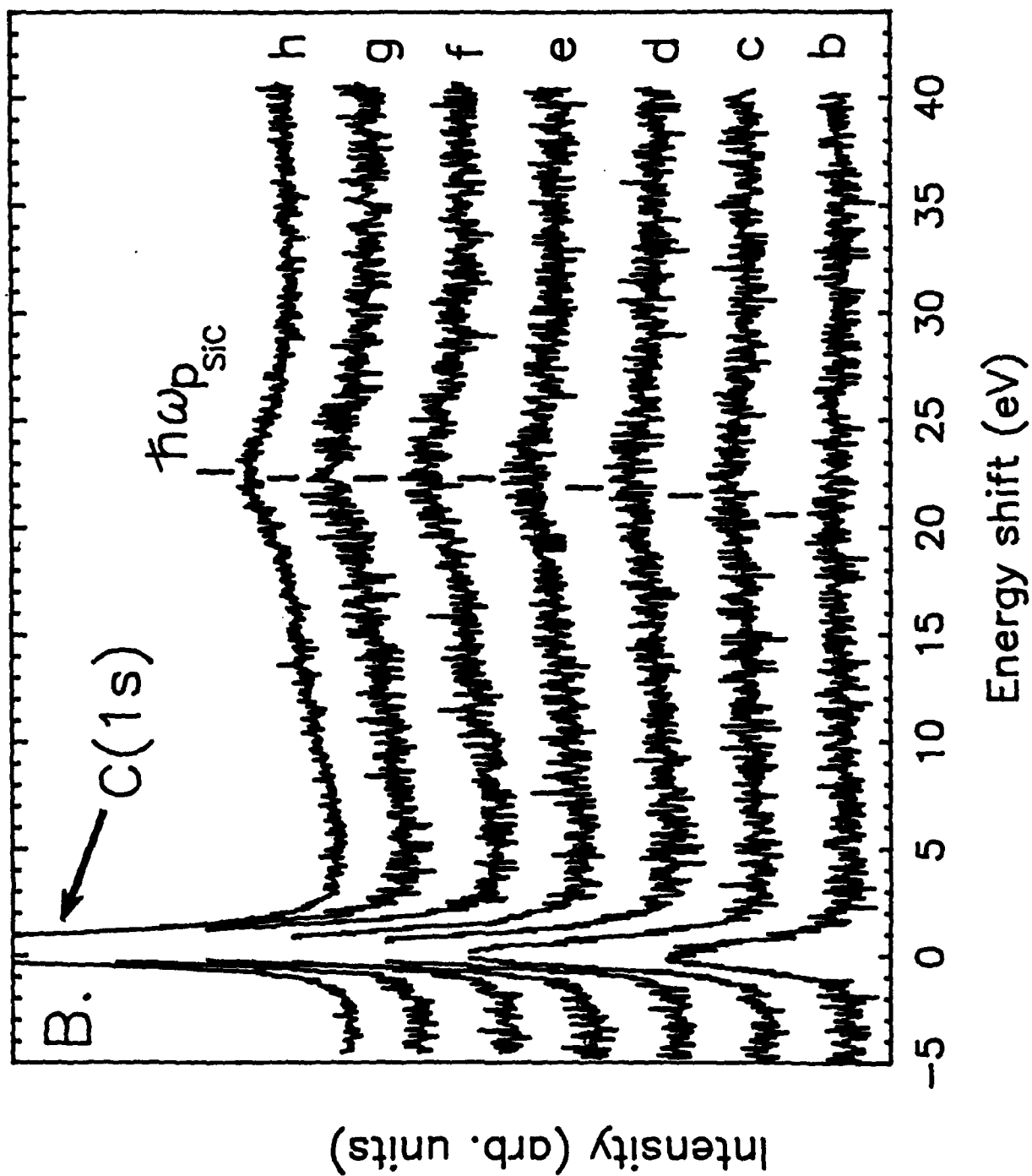




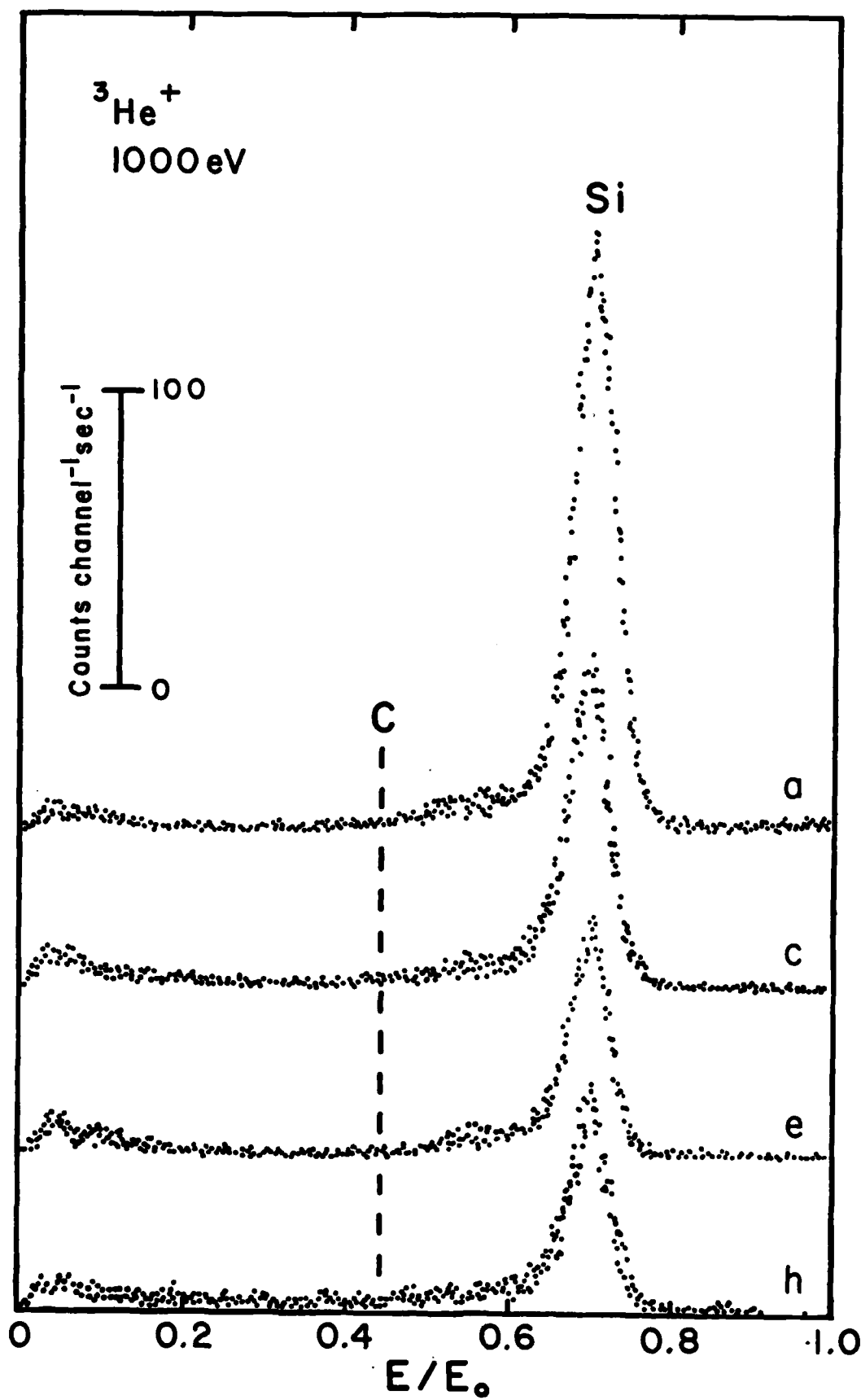
# Development of Plasmon Loss Features — XPS



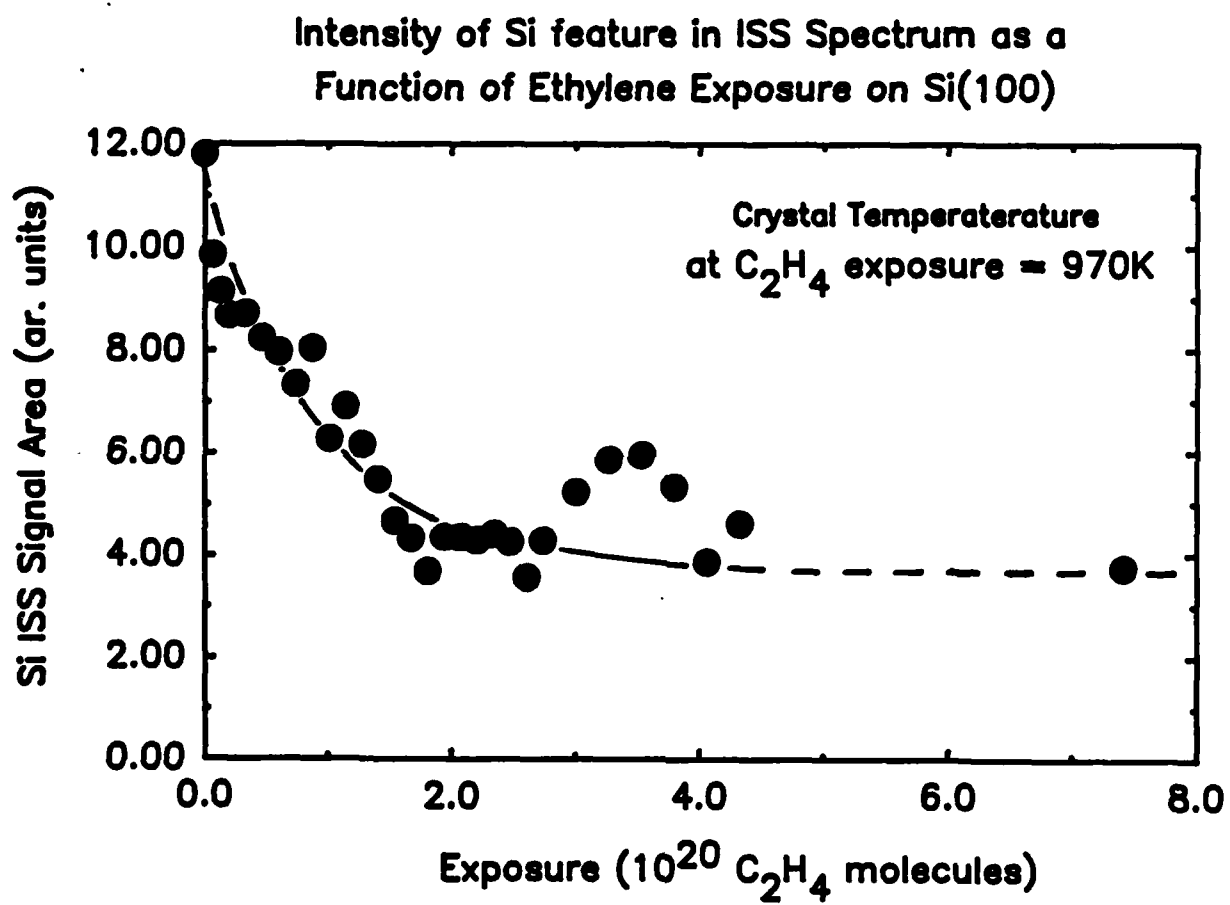
# Development of Plasmon Loss Features - XPS



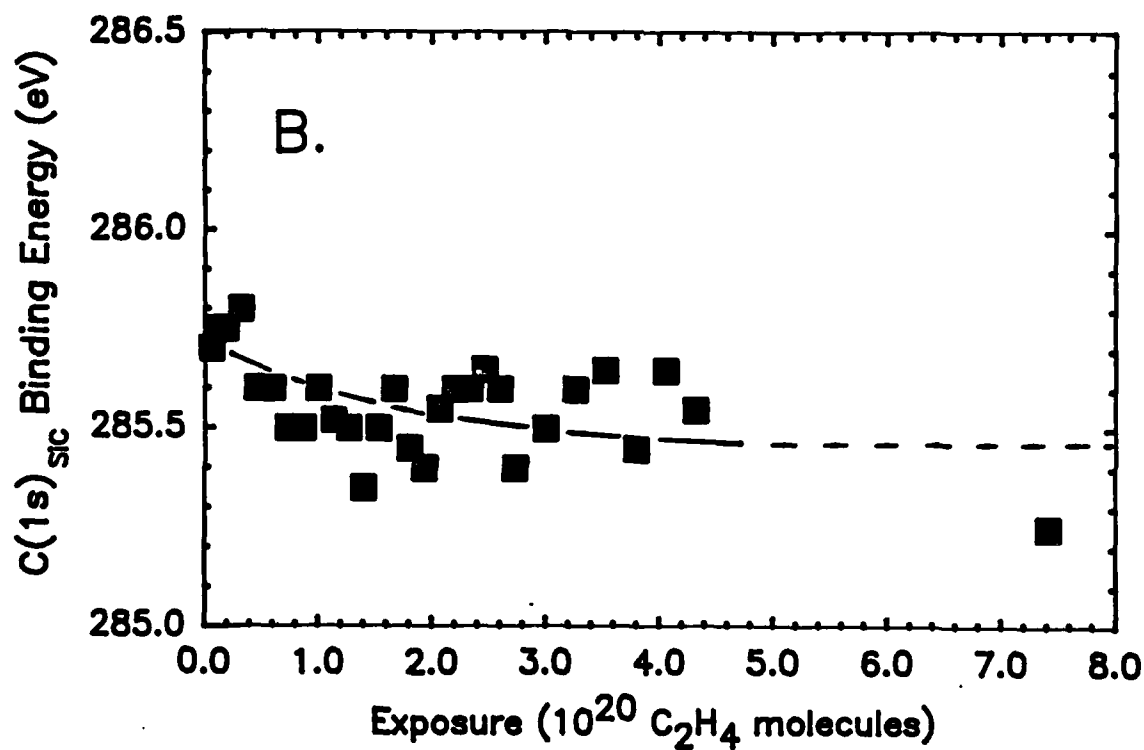
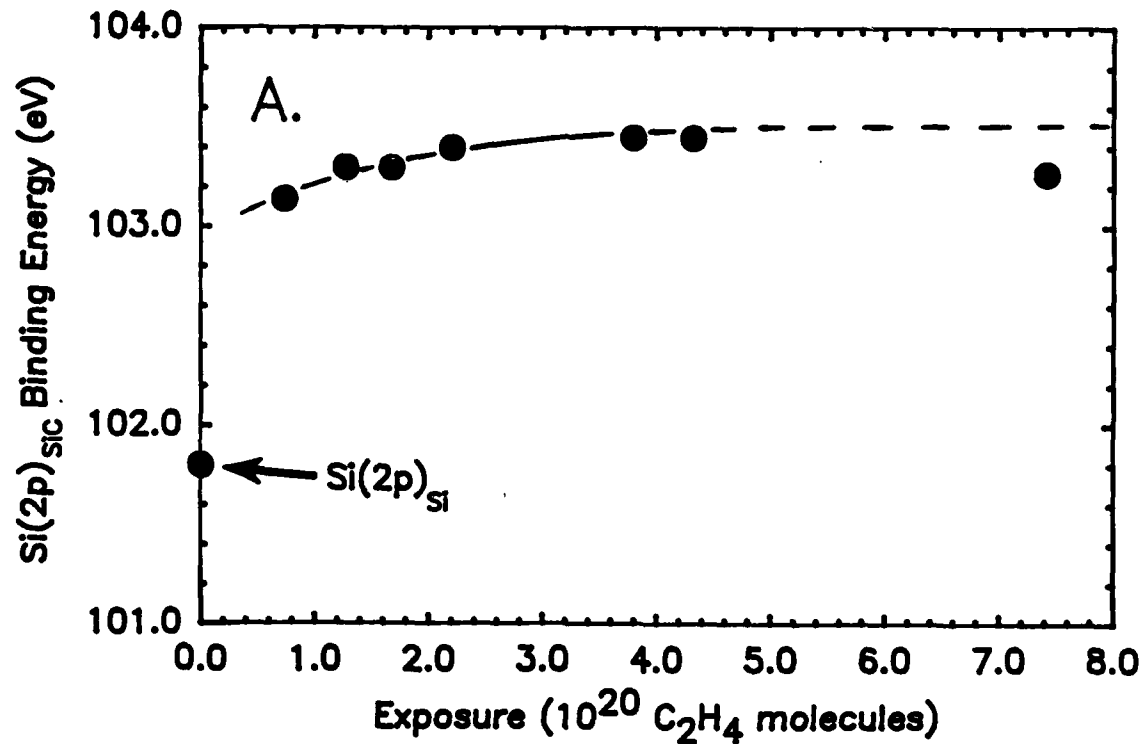
# ISS Behavior During Film Growth on Si(100) at 970K



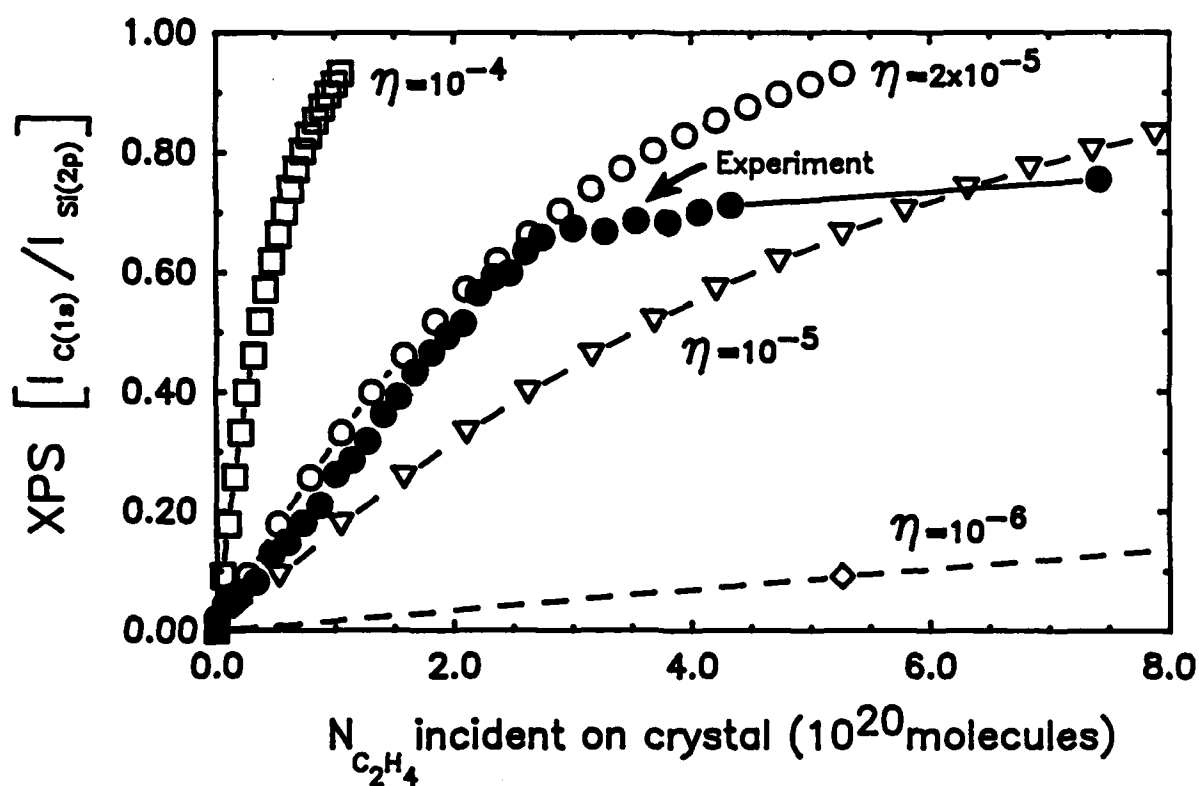
Taylor, et al  
Figure 5



Binding Energies of Si(2p) and C(1s) from SiC  
during CVD growth of SiC on Si(100)



# Efficiency of SiC Production by Ethylene Chemical Vapor Deposition on Si(100) at 970K



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